Presented at John E. Dorn Memorial
Symposium, Fall Meeting AIME,
Cleveland, Ohio, Oct. 16-19, 1972

LBL-1158

HARPER-DORN CREEP OF METALS AT HIGH TEMPERATURES

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September 1972

AEC Contract No. W-7405-eng-48

LBL-1158

DISCLAIMER

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ABSTRACT

This paper reports the current status of research on Harper-Dorn creep: a type of viscous deformation found in some metals and alloys at high temperature and low stress, and characterized by a strain rate which is linearly dependent on applied stress, apparently independent of grain size, and several orders of magnitude above the strain rate obtainable from diffusional processes in large-grained samples. The experimental evidence supporting Harper-Dorn creep in pure aluminum is summarized, along with data indicating the occurance of Harper-Dorn creep in other materials. Available microstructural evidence on the mechanism of Harper-Dorn creep is reviewed: this type of creep is apparently due to the motion of dislocations. While the details of the process are unknown, available data may be plausibly interpreted as due to climb of 'saturated' dislocations. Simple Harper-Dorn creep behavior disappears when the applied stress exceeds a critical value or when sample grain size becomes sufficiently small.

1. INTRODUCTION

Harper-Dorn creep refers to the anomalous creep behavior first observed by Harper and Dorn[1, 2] in their studies on the steady state creep of pure aluminum at very low stresses and temperature near the melting point. The new creep behavior was manifested by several characteristics. The most important of these are: (1) the creep rate is linearly proportional to applied stress; (2) the activation energy for creep is nearly equal to that for self diffusion; (3) single crystals exhibit almost the same creep rate as polycrystals. The first and second characteristics suggested that such a creep behavior might be ascribed to the Nabarro-Herring creep mechanism.[3, 4] However, the third observation was incompatible with the Nabarro-Herring model, since that model predicts negligible creep rate for single crystals.

While the results of Harper and Dorn are well documented, their studies were confined to a single alloy, sample geometry, and test technique. This raises the question of whether Harper-Dorn creep is a significant new mechanism of creep, or perhaps an experimental or material anomaly. Recent research has hence concentrated in four areas: (1) the reproducibility of the Harper-Dorn data when sample geometry and test techniques are varied; (2) the existence of Harper-Dorn creep in other metals and alloys; (3) the mechanism of Harper-Dorn creep; (4) the range of conditions over which Harper-Dorn creep should be observed. We discuss these research areas in turn.

2. HARPER-DORN CREEP IN PURE ALUMINUM

Harper-Dorn creep was first observed in pure aluminum by Harper and Dorn[1], and Harper, Shepard and Dorn[2], who tested thin platelets of both single and polycrystalline aluminum in tension. The thickness (t) of their platelet specimens was approximately 2.5 mm, and the mean grain diameter (d) of their polycrystalline specimens was approximately 3 mm. Although the creep data gave the stress exponent n = 1, and the activation energy for creep was nearly equal to that for self diffusion, the measured creep rate was more than three orders of magnitude greater than predicted by the Nabarro-Herring[3, 4] model for the grain size employed. Moreover, several auxiliary observations, including the observation of almost the same creep rate in a single crystal, the uniformity of strain inside the grain as well as across the brain boundary (marker experiment), and the appearance of primary creep, were inconsistent with the characteristics of Nabarro-Herring creep. The results of Harper and Dorn have been recently confirmed in two distinct investigations. The first was conducted by Barrett, Muehleisen and Nix[5, 6] who tested bulk tensile specimen of both single and polycrystalline ($d \sim 10$ mm) aluminum in tension. In the second investigation, conducted by Mohamed, Murty and Morris[7], bulk double shear specimens of both single and polycrystalline ($d \sim 9$ mm) aluminum were tested in pure shear.

The aggregate results of these three investigations are shown in Fig. 1, where a non-dimensional shear strain rate $(\frac{\dot{\gamma} kT}{DGb})$ is plotted against a non-dimensional shear stress τ/G on log scale. The symbols appearing in these parameters are: $\dot{\gamma}$, the shear strain-rate; k, Boltzman's constant; T, the absolute temperature; D, the self diffusion coefficient; G, the shear modulus; b, the magnitude of the Burgers

vector of a simple dislocation in fcc aluminum lattice; and τ, the effective shear stress. To obtain this plot the tensile data of Harper and Dorn[1, 2] and Muehleisen, et al[5, 6] were transformed to shear data using the relations

$$\dot{\gamma} = \frac{3}{2}\dot{\epsilon}$$

$$\tau = \frac{1}{2}(\sigma - \sigma_o) \qquad [1]$$

where $\dot{\epsilon}$ is the tensile strain rate and σ is the tensile stress. The self-diffusivity D was computed from the relation $D = 0.45 \exp(-33{,}000/RT)$[8]. The value, σ_o, appearing in the second of these relations is a correction term Harper and Dorn found necessary to account for an apparent back stress presumably due to surface effects in thin platelet samples. Since the other two investigations used bulk samples, no correction term is necessary; the data extrapolate naturally to zero strain rate at zero applied stress.

The data of Fig. 1 divide into two regions. The high stress region, labelled II in the figure, shows the characteristic creep behavior generally attributed to climb-dominated dislocation processes. The strain rate in this region is given by the power law

$$\frac{\dot{\gamma}bT}{DGb} = A'(\tau/G)^n \qquad [2]$$

where the stress exponent n is approximately 4.5, as found in several independent investigations.[15] The low stress region, labelled I in Fig. 1, is the domain of Harper-Dorn creep. The data for this creep behavior are well represented by a linear relation

$$\frac{\dot{\gamma}kT}{DGb} = A(\tau/G) \qquad [3]$$

where A is a dimensionless constant approximately equal to $\sim 10^{-10}$.

The scatter about the creep curve in Fig. 1 is due principally to a small consistent deviation between the results of Barrett, et al,[5, 6] and those of the other two studies. The data also scatter somewhat near the knee of the curve where the mechanism of creep is changing. Despite the difference in the kind of specimen used (tensile or shear), the type of stressing (tension or pure shear), and grain size of polycrystalline specimens, the agreement between these three distinct investigations is excellent. For comparison, Fig. 1 also contains a plot of the creep rate predicted from the Nabarro-Herring[3, 4] model for the grain size (9 mm) used in tests by Mohamed, Murty and Morris.[7] Nabarro-Herring creep obeys a linear relation of the form

$$\frac{\dot{\gamma} b T}{D G b} = B \left(\frac{b}{d}\right)^2 (\tau/G) \qquad [4]$$

where B is a dimensionless constant expected to be about 14. As can be seen from the Fig. 1, the predicted rate of Nabarro-Herring creep is roughly three orders of magnitude below the experimental creep rates. Our conclusion from this extensive work on aluminum is that the existence of Harper-Dorn creep in pure aluminum is well established experimentally.

3. EVIDENCE FOR HARPER-DORN CREEP IN OTHER METALS AND ALLOYS

The first clear evidence for Harper-Dorn creep in a material other than pure aluminum was found by Ziling[10] in studies of the creep of silver foils under low stress. The curve through his data is plotted (in our representation) in Fig. 2. The data were taken at a single temperature, 950°C, on foils having thickness $\sim$60μ and grain diameters $\sim$200μ. The creep rates found in region I of this

curve are, in fact, comparable to those predicted from the Nabarro-Herring model for this grain size. However, Ziling obtained direct measurements of grain elongation during creep, using both marker and surface interferometric techniques, and concluded that grain elongation can account for no more than 30% of the total deformation. The remainder must, he concludes, be attributed to deformation in the interior of grains, presumably through creep of the Harper-Dorn type. As can be seen from Fig. 2, the dimensionless creep rates of these specimens are close to those observed in Harper-Dorn creep of aluminum.

More recently evidence for Harper-Dorn creep has been found in an aluminum-magnesium alloy, pure lead, and pure tin. Murty, Mohamed, and Dorn[9] studied the creep of Al-3%Mg. Double-shear specimens of this alloy having a mean grain size of approximately 3 mm were tested over a range of temperatures near the melting point and stresses to approximately $10^{-6}G$. The experimental results are shown schematically in Fig. 2. They reveal a change in creep behavior at $\tau \sim 10^{-5}G$. Below this transitional stress the strain rate depends linearly on the stress and the activation energy for creep approximates that for self-diffusion. The creep rates observed in region I are more than two orders of magnitude above those predicted on the basis of the Nabarro-Herring model (Table I). Moreover, normal primary creep is evident in tests conducted within region I. These results, coupled with the observation that the observed dimensionless creep rates in region I are reasonably close to those found for pure aluminum, lead to the conclusion that Harper-Dorn creep is dominant in Al-3%Mg at low stress.

More recently, low stress, high temperature creep studies have been conducted on lead and tin.[7] These two metals were chosen because

of their low melting points (allowing good temperature control in tests at temperatures near the melting point) and their differing crystal structures (lead is face-centered cubic, tin is body centered tetragonal). The results of these tests are similar to those obtained in the studies of Al and Al-3% Mg. Tests over a range of temperatures and stresses yield composite creep curves (shown in Fig. 2) which divide into two distinct regions separated by a knee.

In the case of lead, creep behavior in region II obeys a relation of the form [2] with stress exponent (n) equal to approximately 4.9; creep behavior in region I obeys a linear relation between stress and strain rate (Table I). The activation energy for creep in both regions is approximately the activation energy for self-diffusion. The creep rates in region I lie considerably above those predicted from the Nabarro-Herring model for the grain size (1.5 mm) of the lead samples used (Table I), but are within a factor of about three of the dimensionless creep rates found in the Harper-Dorn region of pure aluminum. In addition, lead undergoes an appreciable primary creep at all stresses used.[7]

The creep behavior of tin is qualitatively similar to that of lead though its stress exponent in region II is considerably larger, $n \sim 6.6$ as compared to $n \sim 4.9$ for lead (Table I). The creep curve of tin (Fig. 2) again shows a "knee" at $\tau \sim 10^{-5}G$, $\dot{\gamma} \sim 10^{-15}$ (DGb/kT) and a low stress region I obeying a nearly linear relation between stress and strain-rate. The activation energy for creep in region II agrees well with the self-diffusivity of tin[(7)] (which is measured as a phenomenological average between the longitudinal and transverse diffusivities of the anisotropic BCT tin lattice). The activation energy for creep in region I is assumed to be the same, but has not been checked since the very low absolute strain rates in region I

of tin have restricted measurements to $T > 0.98T_m$, giving an insufficient temperature range to determine valid activation energies. The strain rates obtained in region I for tin are uniformly more than two orders of magnitude above the Nabarro-Herring prediction for the grain size (2 mm) of the samples used, but are very close to the (non-dimensional) strain rates obtained in Harper-Dorn creep of aluminum. Normal primary creep is observed at all stresses used.

Hence there is now persuasive evidence for Harper-Dorn creep in four materials other than pure aluminum: Al-3Mg, silver, lead and tin. To make this evidence definitive one should demonstrate that the strain rates in region I of these materials are strictly independent of grain size. Previous work indicates this lack of dependence, since small variations in mean grain size from sample to sample have no apparent effect on the creep rates obtained. Attempts to gather more definitive data have been complicated by sample preparation problems and by recrystallization problems in high temperature tests on very large grained samples of lead and tin.

The apparent success in finding Harper-Dorn creep in other materials suggests that Harper-Dorn creep may be an important general mechanism of creep at high temperature and low stress. However, not all attempts to find Harper-Dorn creep behavior have been successful. Muehleisen[5] saw no evidence of Harper-Dorn creep in studies of the creep of compressive specimens of copper at low stress. His measurements extended down to stresses $\tau \sim 5.10^{-6}G$ and non-dimensional strain rate $\frac{\dot{\gamma}kT}{DGb} \sim 10^{-16}$. As can be seen from Fig. 2, this range of conditions extends slightly into the range of apparent Harper-Dorn creep in other materials. Pines and Sirenko[11] may have observed Harper-Dorn creep in copper at $\tau \sim 5 \times 10^{-6}G$, but the evidence is not clear.

4. THE MECHANISM OF HARPER-DORN CREEP

The available evidence does not require that there be a single mechanism of Harper-Dorn creep; there may be several. However, the available data may be plausibly explained as due to creep via climb of "saturated" dislocations, one of two mechanisms suggested by Friedel[12] as natural modifications of the Nabarro-Herring mechanism in a dislocated crystal. Other candidate mechanisms have been suggested. These require more restrictive assumptions and are hence less appealing hypotheses.

The body of available microstructural evidence, which is unfortunately drawn almost entirely from research on pure aluminum, indicates that Harper-Dorn creep is dislocation-dominated and characterized by a low density of dislocations dispersed through rather large subgrains. Evidence for a dislocation-dominated mechanism was noted in the original work of Harper and Dorn[1] and Harper, Shepard and Dorn.[2] This evidence includes the observation of strain recovery following creep, the measurement of strains within individual grains comparable to those across grain boundaries, and the occurance of extensive primary creep. More recent work by Muehleisen[5] and by Barrett, Muehleisen, and Nix[6] established subgrain formation during Harper-Dorn creep of aluminum and showed that the rate of Harper-Dorn creep can be significantly reduced by distributing a fine precipitate (in this case Al_3Fe) through the microstructure. Ziling[10] concluded that 70-90% of the total strain he observed during low-stress creep of silver was due to dislocation motion in the interior of grains.

Available evidence indicates that the dislocation density (ρ) during Harper-Dorn creep of aluminum is low ($\leq 10^4$ cm^{-2}) and independent

of the applied stress. This result was obtained both by Muehleisen[5] from etch pit counts, and by Nost and Nes[13], who used x-ray topographic techniques. The data are plotted in Fig. 3 as the dimensionless parameter $\sqrt{\rho}b$. The evidence on subgrain formation is less definitive. Harper, Shepard and Dorn[2] did not observe subgrains, probably because of sample handling technique (J. E. Dorn, private communication). Muehleisen[5] noted subgrains and obtained data on the variation of subgrain size with applied stress. The stress data are ambiguous (compare refs. 5 and 6); however, the subgrains are large, having diameters in the range 1-3 millimeters. The dislocations are distributed generally through these subgrains. Ziling[10] studied etched surfaces of silver deformed in Harper-Dorn creep. While he does not report dislocation densities, his published micrograph shows a low, rather uniform dispersion of dislocations.

These observations are in good agreement with a creep mechanism dominated by climb of dislocations under "saturated" conditions, that is, when the dislocation velocity due to climb is controlled by the rate at which vacancies can diffuse to (or from) the dislocation line. The consistency of this mechanism with the experimental data may be summarized as follows. (1) The strain rate due to creep at low stress is given, according to Hirth and Lothe,[14] by an equation which may be rewritten in the form of the governing equation [3] of Harper-Dorn creep with

$$A_{HD} = -\frac{6\pi\rho b^2}{\ln(\sqrt{\rho}b)}. \qquad [5]$$

Given $\rho \simeq 10^4$ cm^{-2} and $b = 2.86 \times 10^{-8}$ cm for aluminum we compute $A_{HD} = 1.207 \times 10^{-11}$, in reasonable agreement with the experimental value, $A_{HD} = \sim 10^{-10}$. (2) At low stress the activation energy for creep via this mechanism is the activation energy for self

diffusion. (3) The mechanism is dislocation-dominated. Its rate should be markedly decreased if a fine precipitate is dispersed through the matrix. (4) The rate of creep via this mechanism will be independent of grain size for large grain sizes. (5) The low dislocation densities observed during Harper-Dorn creep are sufficient to saturate any dislocation which contains a preponderance of jogs of one type (vacancy-absorbing or vacancy-emitting). Friedel[12] predicts saturation for dislocation densities $\rho \gtrsim 10^5$ cm^{-2}.

Several other suggested mechanisms of Harper-Dorn creep also show promising agreement with the experimental data, though they give less appealing models. These include creep through glide of jogged screw dislocations, creep through unsaturated climb of jogged edge dislocations, and creep of the Nabarro-Herring type within subgrains.

Creep through the glide of jogged screw dislocations was the mechanism favored by Harper, Shepard and Dorn.[2] By modifying the equation resulting from more recent studies, as given by Hirth and Lothe[14], we obtain an equation of the form [3] with

$$A_{js} = 12\pi\rho\lambda b, \qquad [6]$$

where λ is the spacing between jogs. Equation [6] gives a creep rate of the order observed if $\lambda \sim b$, an extremely high jog density. If these jogs are of the same type, as suggested in earlier work by the present authors[5], the dislocation will certainly saturate. If the jogs are of opposite type they will tend to annihilate by climb, and will lead to an overall activation energy for creep which is expected[14] to show the effects of core diffusion. In any case such high jog densities seem inconsistent with the assumptions of the model leading to equation [6].

Creep through the climb of jogged edge dislocations is governed by[14] an equation of the form [3] with

$$A_{JE} = 12\pi\rho b^3/\lambda \qquad [7]$$

equation [7] predicts a creep rate near that observed if $\lambda \sim 20b$, which is roughly the magnitude expected for thermal jogs in aluminum near the melting point.[5] However, the assumption of a large population of nearly straight edge dislocations near the melting point is difficult to accept, and again a dislocation containing a jog density is low. If the dislocation did not saturate one would expect[14] core diffusion to influence the activation energy for creep, in apparent conflict with the experimental data.

Friedel[12] originally favored a Nabarro-Herring mechanism acting across subgrains as the mechanism of Harper-Dorn creep. This mechanism obeys an equation of the form [4] with the parameter d equal to the mean subgrain size. As several authors have pointed out[5, 9], the large subgrain size observed during Harper-Dorn creep of aluminum cause the creep rate predicted by this mechanism to fall well below that actually observed. Moreover, the subgrain size in aluminum either decreases with applied stress[6] or at least shows appreciable scatter over the domain of Harper-Dorn creep.[5] This variation would induce a scatter in measured creep rate considerably greater than that observed experimentally.

Finally, Barrett, Muehleisen, and Nix[6] have recently suggested a model based on a balance between the generation and annihilation of gliding dislocations. The model is intriguing, but is not yet sufficiently developed for criticism.

5. THE DOMAIN OF HARPER-DORN CREEP

Harper-Dorn creep is limited to stresses and strain rates below the knee of the material creep curve. In the absence of established creep mechanisms the position of this knee cannot be predicted. However, if we speculate that this knee is due to a change in dislocation density from the low, constant value found in region I to the high, increasing values ($\rho \propto \tau^2$) found in region II[17], two interesting correlations may be drawn. First, let the change in dislocation density be due to activation of dislocation sources at a critical value of the applied stress, $(\tau/G)_c$. Let these sources be nodes in the array of dispersed dislocations, having an expected spacing $(\rho)^{-1/2}$, the characteristic length of the array. Then dislocation multiplication by the bow-out mechanism[12] gives

$$(\tau/G)_c \simeq 2\sqrt{\rho}\, b, \qquad [8]$$

or 5.6×10^{-6} for aluminum having $\rho \sim 10^4 cm^{-2}$, very close to the value observed. Alternatively, let the total dislocation density be the sum of a residual density $\rho_o \sim 10^4 cm^{-2}$ and a stress-dependent density which increases according to the relation

$$\rho' = \left(\frac{1}{\alpha b}\right)^2 (\tau/G)^2 \qquad [9]$$

where α is a constant of order 1.0, as observed in the creep of several metals at higher stress.[17] We have plotted equation [9] for three values of the parameter α in Fig. 3. The extrapolated curves intersect the measured dislocation density for aluminum at stresses near the knee of the creep curve.

Recent data indicates that Harper-Dorn creep is also limited by sample grain size. Burton[15] studied the low-stress, high temperature creep of fine-grained aluminum foils. He measured creep rates

which may be shown[16] to obey an equation of the form [4] of the governing equation of Nabarro-Herring creep with B ∿ 130. His data are plotted together with the creep curve for large-grained aluminum in Fig. 4. The measured creep rates are uniformly higher than those observed in Harper-Dorn creep of bulk aluminum.

This effect of small grain size is anticipated on the basis of any one of the candidate mechanisms discussed in section 4. These competing mechanisms may increase the rate of Nabarro-Herring creep at small grain size by providing vacancy sources and sinks which decrease the effective diffusion distance, but they cannot decrease the Nabarro-Herring creep rate. Since the rate of Nabarro-Herring creep increases with d^{-2}, where d is grain size, one must anticipate a value of d, d_c, below which a creep of the Nabarro-Herring type is observed. Reasoning from Burton's data, $d_c \sim 640\mu$ in aluminum. The creep rates measured by Burton on fine grained samples are above those predicted from the Nabarro-Herring relation by a factor of about 6. This discrepancy may be due to the approximations of the model, or may show the influence of dislocations in the interior of the grains.

ACKNOWLEDGEMENT

This work is the outgrowth of research inaugurated and conducted by Professor John E. Dorn. Much of the work reported was done under his supervision. The remainder shows his strong influence.

This work was supported by the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory.

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TABLE CAPTIONS

Table I. Values of (A) and (N) obtained from the least squares analyses of creep data for materials which show apparent Harper-Dorn creep. The values predicted on the assumption of Nabarro-Herring creep are included.

TABLE I

Metal	Region I (Harper-Dorn)		Region II (climb)		Nabarro	
	N	A	N	A	N	A
Aluminum (d = 9 mm)	1.07 ± 0.07	$(3.72 \pm 5.62 / 3.57) \times 10^{-10}$	4.5	10^{8}	1	1.42×10^{-14}
Aluminum-3% Magnesium (d = 3 mm)	0.91 ± 0.03	$(2.88 \pm 1.39) \times 10^{-11}$	4.05 ± 0.07	$(1.79 \pm 1.50) \times 10^{5}$	1	1.26×10^{-13}
Lead (d = 1.5mm)	0.97 ± 0.08	$(2.88 \pm 4.88 / 1.81) \times 10^{-11}$	4.92 ± 0.57	$(1.38 \pm 405.62 / 1.375) \times 10^{8}$	1	8×10^{-13}
Tin (d = 2 mm)	0.94 ± 0.07	$(7.24 \pm 9.76 / 4.22) \times 10^{-11}$	6.59 ± 0.28	$(3.55 \pm 59.55 / 3.35) \times 10^{17}$	1	3.15×10^{-13}
Silver (d = 0.2mm)	1	6.6×10^{-11}	UNCERTAIN		1	2.95×10^{-11}

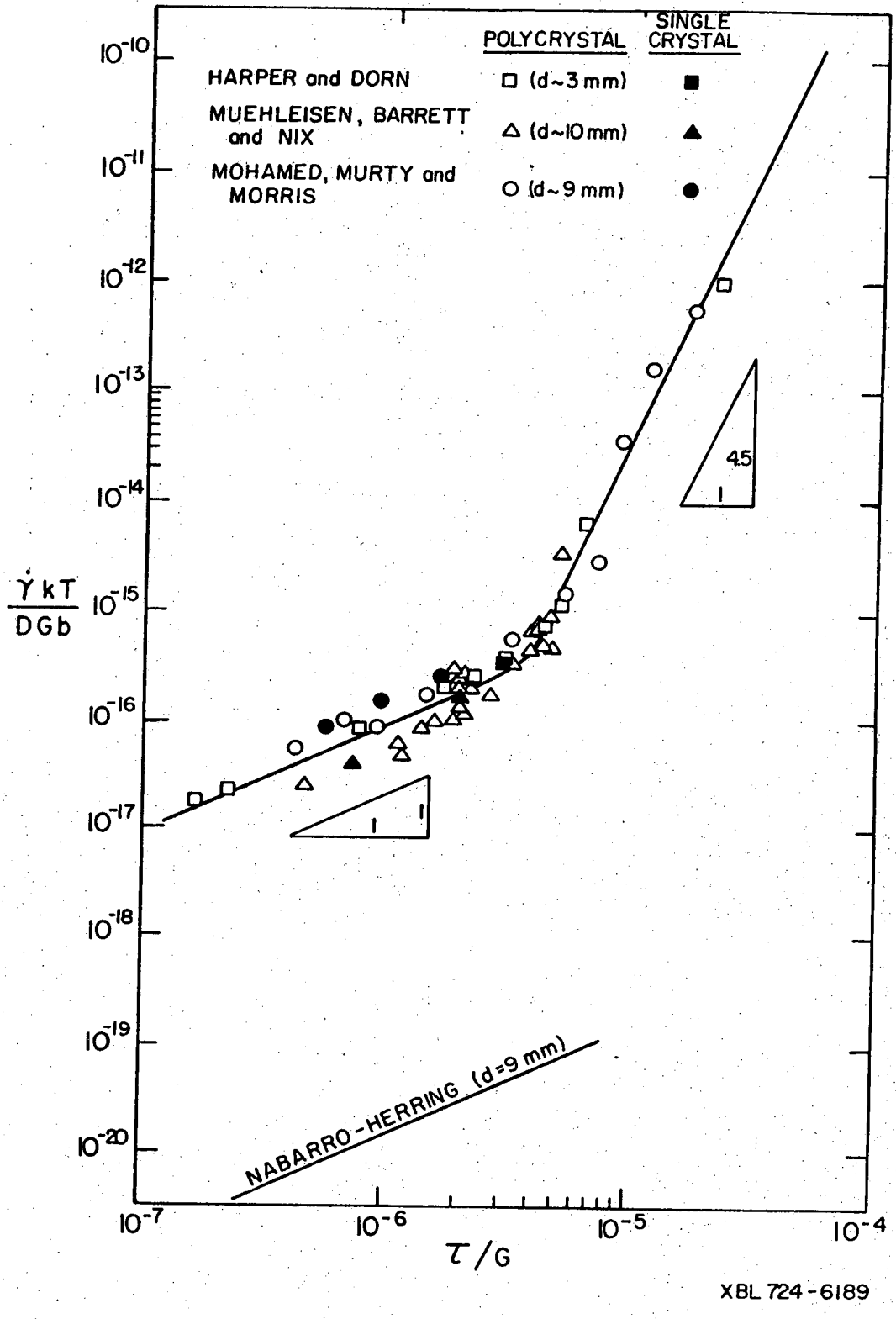
FIGURE CAPTIONS

Fig. 1 Data of three investigations of the low-stress, high temperature creep of coarse-grained aluminum.

Fig. 2 Data on creep of tin, lead, aluminum - 3% magnesium and silver compared to creep behavior of aluminum. Diffusivity date are from Ref: Al (8), Sn(18), Pb(19), Al-3%Mg(9) and Ag(20).

Fig. 3 Stress dependence of dislocation density.

Fig. 4 The data of Burton on the creep of fine-grained foils of aluminum in vacuum (data points) compared to creep behavior of aluminum in bulk samples (solid line). The value of the self-diffusivity, D, is taken from the relation proposed by Burton.[15]


POLYCRYSTAL
SINGLE CRYSTAL
HARPER and DORN
□ (d~3 mm)
■
MUEHLEISEN, BARRETT and NIX
△ (d~10 mm)
▲
MOHAMED, MURTY and MORRIS
○ (d~9 mm)
●
45
1
1
1
NABARRO-HERRING (d=9 mm)
$\frac{\dot{\gamma} kT}{DGb}$
10^{-10}
10^{-11}
10^{-12}
10^{-13}
10^{-14}
10^{-15}
10^{-16}
10^{-17}
10^{-18}
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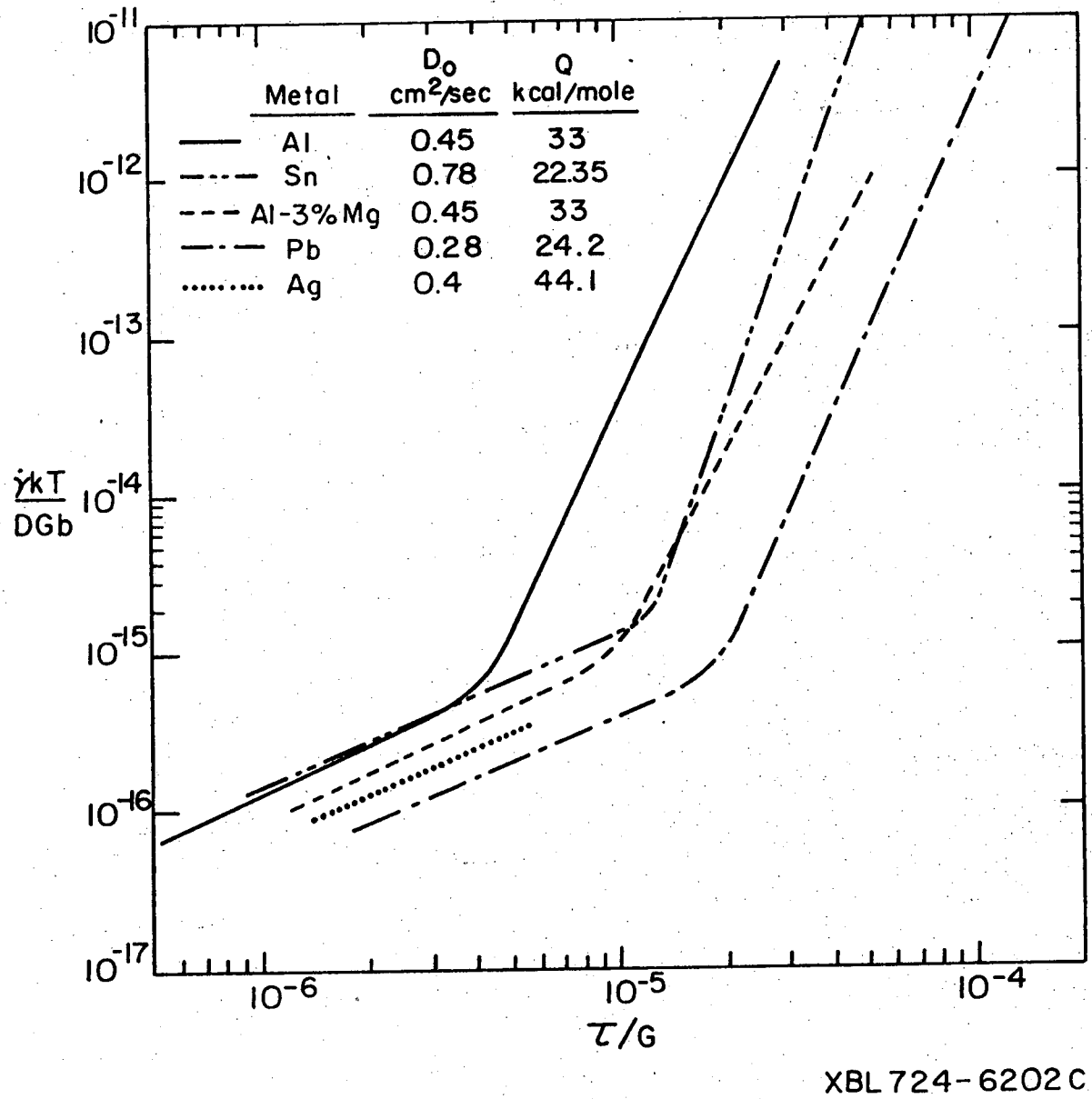
10^{-4}
τ/G
XBL 724-6189

Fig. 1


Metal
D_o cm²/sec
Q kcal/mole
Al 0.45 33
Sn 0.78 22.35
Al-3%Mg 0.45 33
Pb 0.28 24.2
Ag 0.4 44.1
$\frac{\dot{\gamma}kT}{DGb}$
10^{-11}
10^{-12}
10^{-13}
10^{-14}
10^{-15}
10^{-16}
10^{-17}
10^{-6}
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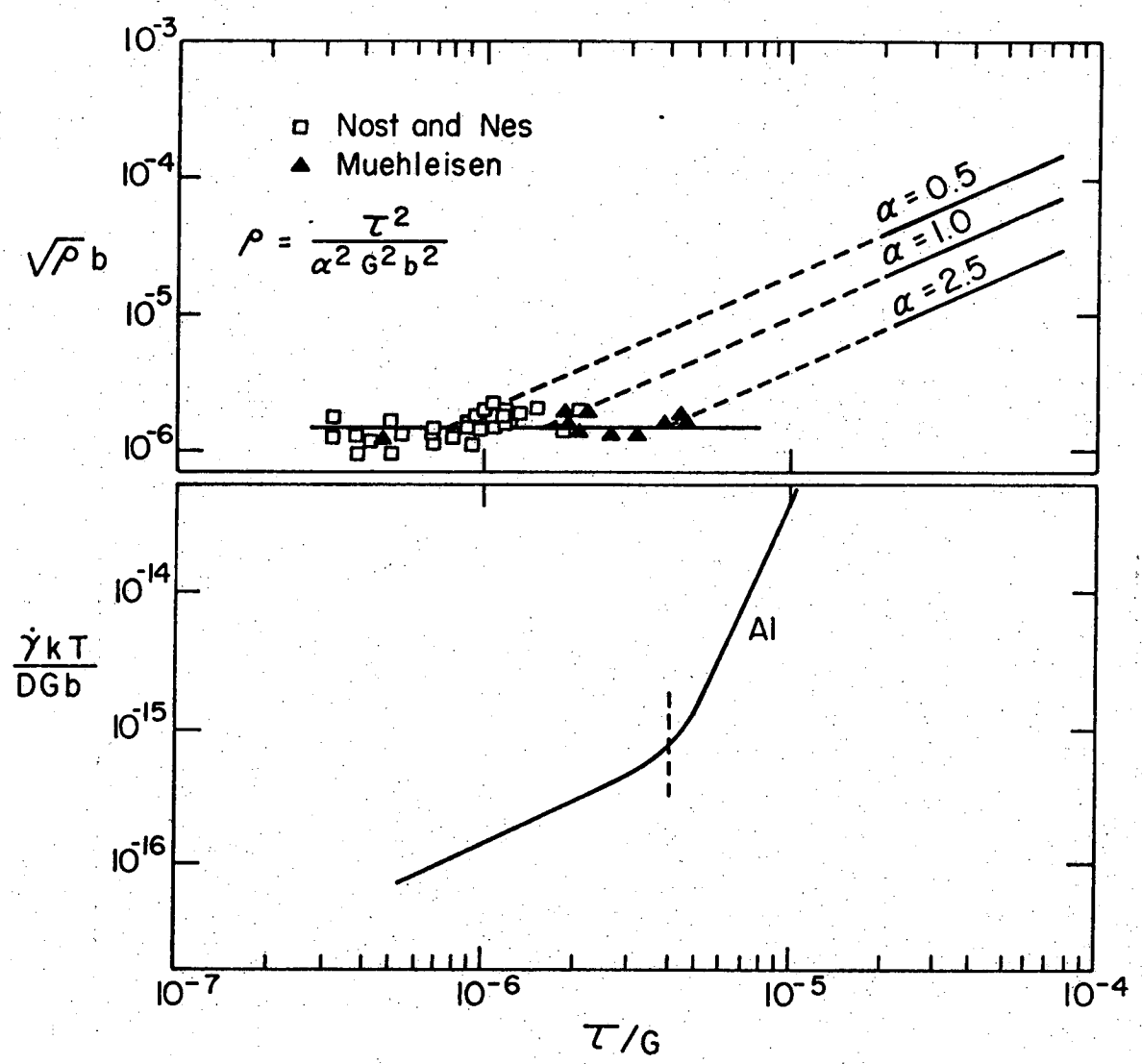
10^{-4}
τ/G
XBL 724-6202C

Fig. 2



XBL 724-6204A

Fig. 3

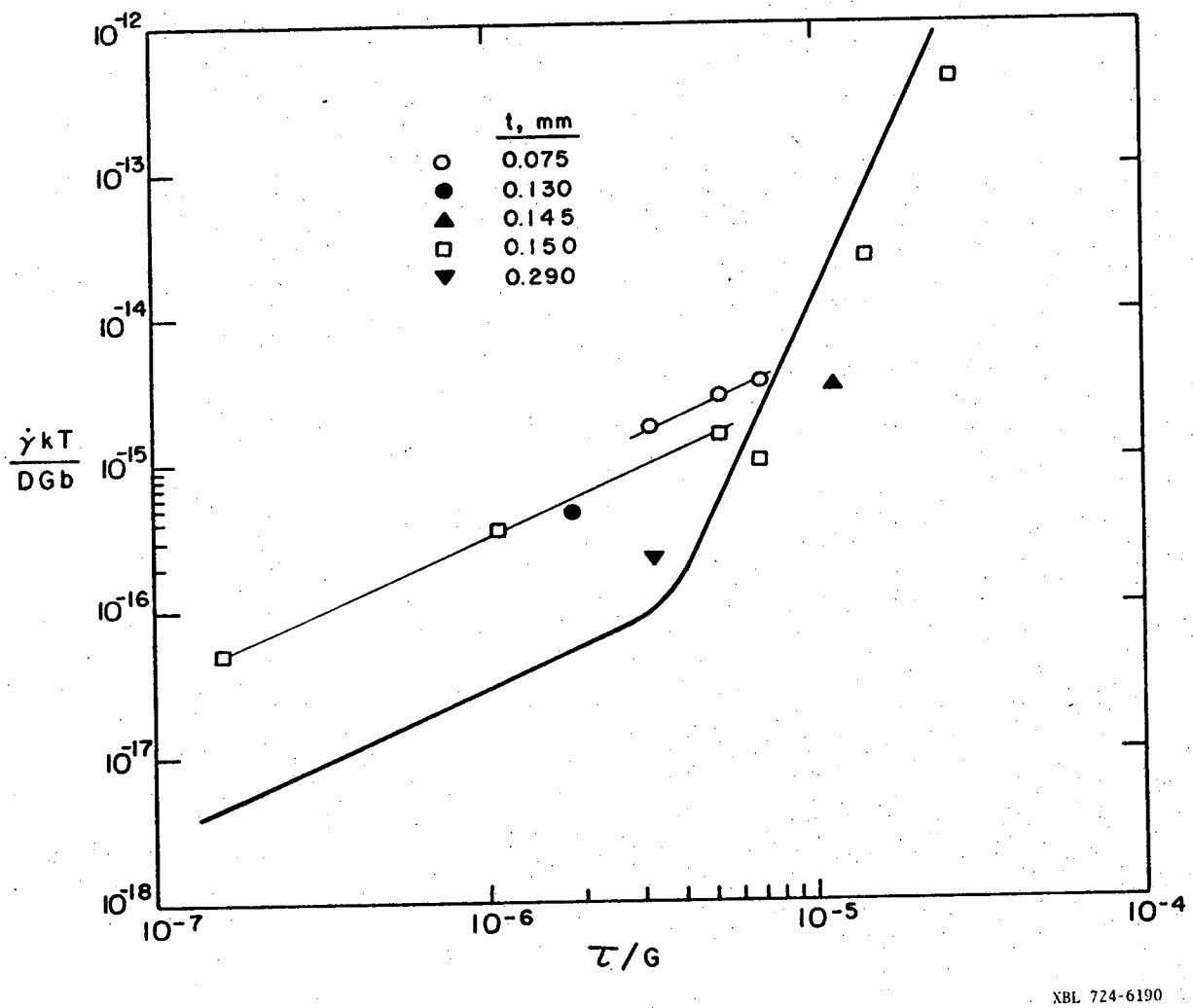

t, mm
0.075
0.130
0.145
0.150
0.290
$\frac{\dot{\gamma} kT}{DGb}$
10^{-12}
10^{-13}
10^{-14}
10^{-15}
10^{-16}
10^{-17}
10^{-18}
10^{-7}
10^{-6}
10^{-5}
10^{-4}
τ/G
XBL 724-6190

Fig. 4

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